

## Design of very transparent fluoropolymer resists for semiconductor manufacture at 157 nm

A.E. Feiring<sup>\*</sup>, M.K. Crawford, W.B. Farnham, J. Feldman, R.H. French, K.W. Leffew, V.A. Petrov, F.L. Schadt III, R.C. Wheland, F.C. Zumsteg

DuPont Central Research and Development, Experimental Station E328/231, P.O. Box 80328, Wilmington, DE 19880-0328, USA

### Abstract

Photolithography at 157 nm requires development of new photoresists that are highly transparent at this wavelength. Transparent fluoropolymer platforms have been identified which also possess other materials properties required for chemically amplified imaging and aqueous development. Polymers of tetrafluoroethylene (TFE), a fluoroalcohol-substituted norbornene and an acid-labile acrylate ester show the best combination of properties. A solution, semibatch, free-radical polymerization process was developed allowing synthesis of the terpolymers on a multikilogram scale. Further property enhancements may arise from replacing the norbornene with functionalized tricyclonones. Formulated resists have been imaged in a 157 nm microstepper.

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### 1. Introduction

#### 1.1. Photolithography

The astonishing advances in computer technology over several decades derive largely from the continuing development of smaller and faster computer chips. This development has been enabled by use of ever smaller features on the chips, going from micron-sized to about 120 nm at the current state-of-the-art for critical features.

The complex patterns on a computer chip are generated by photolithography [1] in which light of a given wavelength is passed through a mask containing the desired pattern, focused and allowed to impinge on a photoresist applied as a thin film on the wafer substrate surface. The light causes a chemical change in the photoresist which (in a positive working resist) renders the material more soluble so that the irradiated areas can be removed by washing with a given solvent. The wafer with the patterned photoresist on its surface is then subjected to an ion-etching process. In the unprotected areas, the underlying substrate is etched whereas the photoresist prevents etching in the imaged areas. After removal of the remaining photoresist, the overall result is that the pattern originally present in the mask has

been faithfully reproduced on the chip surface. This process is repeated multiple times in the construction of the hundreds of million transistors on modern computer chips.

Because minimum achievable feature size is directly proportional to the imaging wavelength, the decrease in feature size has been enabled by the development of imaging technology using shorter wavelengths of light. Thus, leading edge chip manufacture has gone from using 365 nm (Hg i-line) to 248 nm (KrF laser) to the emerging 193 nm (ArF laser) light for imaging. Modern optics allow generation of feature sizes roughly one-half the size of the imaging wavelength. It is anticipated that the next step in this development will be to use of light at 157 nm, provided by the F2 laser, which should emerge as a commercial process in the middle of this decade.

#### 1.2. Photoresist technology

A modern photoresist contains multiple components but the most important is a polymeric binder. The binder polymer must have many critical properties [2] including:

- functionality for image formation and subsequent dissolution;
- transparency at the imaging wavelength so that sufficient light can penetrate through its full thickness;
- etch resistance;

<sup>\*</sup> Corresponding author. Tel.: +1-302-695-1841; fax: +1-302-695-9799. E-mail address: [andrew.e.feiring@usa.dupont.com](mailto:andrew.e.feiring@usa.dupont.com) (A.E. Feiring).

- high contrast;
- a glass transition temperature ( $T_g$ ) above typical processing temperatures so that flow does not occur;
- solubility in selected organic solvent for application by spin coating;
- molecular weight sufficiently high for acceptable mechanical properties but low enough for rapid dissolution in developer;
- ability to stick to the silicon or other substrates; and
- extremely high purity, usually measured in parts per billion levels.

The photoresist binder polymer must, of course, also be manufacturable on appropriate scale at an acceptable cost.

Lithography at 248 nm is based on a chemically amplified photoacid-catalyzed process (Fig. 1). The binder polymer is generally poly(*para*-hydroxystyrene) in which some hydroxyls are protected as acid-labile *tert*-butyl carbonates [3]. The protected polymer is insoluble in aqueous base. Irradiation causes decomposition of a photoacid generator (PAG), typically a triarylsulfonium perfluoroalkylsulfonate, to generate strong acid in the irradiated areas. During a post exposure bake step, the acid catalyzes removal of sufficient protecting groups to render the exposed areas soluble in aqueous base for washout. A key feature is that the deprotection is catalytic in protons so one photon can cause many deprotection steps. This chemical amplification of the light is especially important at shorter wavelengths due to the cost of generating high energy photons.

The hydroxystyrene polymers used at 248 nm have another important feature: high etch resistance due to the presence of the aromatic rings. Carbon-rich organics tend to have better etch resistance than materials with larger amounts of hydrogen or oxygen [4].

However, aromatic polymers cannot be used at shorter wavelengths due to their high absorbances. For 193 nm photolithography, several resist polymers have been developed, based on norbornene/maleic anhydride copolymers or poly(meth)acrylates having polycyclic (e.g. adamantyl) groups in their side chains [5]. The polycyclic groups are suitably transparent at 193 nm and provide etch resistance. Typically, *tert*-butyl (meth)acrylate or related groups are incorporated to provide the acid-labile solubility switch.

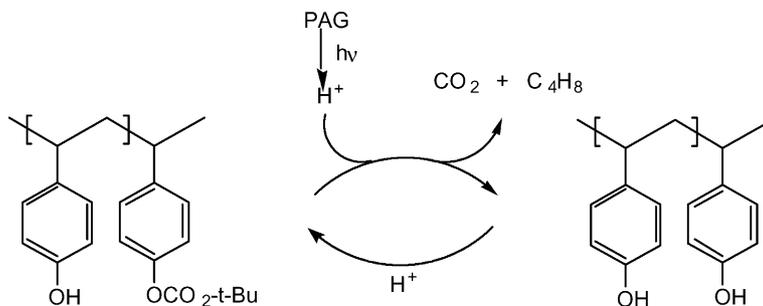


Fig. 1.

Table 1  
Absorption coefficients at 157 nm for selected polymers<sup>a</sup>

| Polymer                     | A ( $\mu\text{m}^{-1}$ ) |
|-----------------------------|--------------------------|
| Poly(hydrosilsequioxane)    | 0.06                     |
| Amorphous perfluoropolymer  | 0.70                     |
| Poly(dimethylsiloxane)      | 1.61                     |
| Poly(vinyl alcohol) (99.7%) | 4.16                     |
| Poly(methylmethacrylate)    | 5.69                     |
| Poly(vinylphenol)           | 6.25                     |
| Poly(norbornylmethacrylate) | 6.73                     |
| Poly(norbornene)            | 6.80                     |
| Poly(acrylic acid)          | 11.00                    |

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At 157 nm the situation becomes even more severe. Although the maximum tolerable absorbance of a resist binder polymer is still under discussion, most agree that it must be under about  $2 \mu\text{m}^{-1}$  and preferably under  $1 \mu\text{m}^{-1}$ . Studies showed that known resists are far too absorbing for use at 157 nm at a reasonable film thickness (Table 1) [6,7]. Styrenic and (meth)acrylate polymers are above  $6 \mu\text{m}^{-1}$ , orders of magnitude higher than the goal value. Interestingly, even the homopolymer of norbornene which is devoid of unsaturated groups was found to have an absorbance of  $6.8 \mu\text{m}^{-1}$ .

### 1.3. Role of fluoropolymers

Two classes of polymers with low absorbance at 157 nm were identified: certain fluoropolymers and polysiloxanes [7]. In particular, Teflon<sup>®</sup> AF, an amorphous copolymer of tetrafluoroethylene (TFE) and perfluoro(2,2-dimethyldioxole), was found to have an absorbance of about  $0.7 \mu\text{m}^{-1}$  at 157 nm. We found that the alternating copolymer of TFE and norbornene has an absorbance of  $1.3 \mu\text{m}^{-1}$  at 157 nm, some five orders of magnitude more transparent than the norbornene homopolymer [8]. This polymer is amorphous, has a desirable  $T_g$  of about  $150^\circ\text{C}$  and was shown to have good etch resistance, comparable to existing photoresists. Our work has since focused on turning the TFE/norbornene copolymer into a fully-functional 157 nm photoresist [9–12]. This paper will be focused on synthesis of the

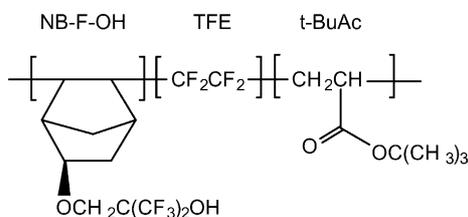


Fig. 2.

resist polymers; properties relating to their lithography performance have been described elsewhere [11].

## 2. Results and discussion

### 2.1. Resist polymer structure

Consideration of the properties needed in a polymer for a 157 nm photoresist led to design of a terpolymer (Fig. 2) of tetrafluoroethylene, *tert*-butyl acrylate (*t*-BuAc) and a norbornene with a pendant fluoroalcohol group. Each component provides one or more of the required properties, but may impart certain disadvantages as well. The carbon-rich norbornane ring provides etch resistance but, as noted above, must be spaced by TFE units for good transparency at 157 nm. The *t*-BuAc units provide acid-labile solubility switches because they are rapidly cleaved to isobutylene and polymer bound carboxylic acids when heated in the presence of photogenerated acid. However, the carbonyl groups are also highly absorbing at 157 nm. We found that incorporation of sufficient *t*-BuAc groups to make the hydrophobic TFE/norbornene polymer soluble in aqueous base after imaging increased the polymer absorbance to above  $3.0 \mu\text{m}^{-1}$ . Hence the need for the fourth component, the pendant hexafluoroisopropanol groups. These are introduced as side chains on the norbornene monomer, NB-F-OH (Fig. 3), and help to provide solubility in aqueous base.

The hexafluoroisopropanol group is known to be as acidic as phenol so it readily ionizes for dissolution in aqueous base

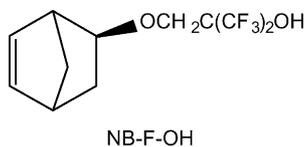


Fig. 3.

(hexafluoroisopropanol-substituted polymers have been used for photolithography at longer wavelengths [13]). In contrast to the phenols, however, the hexafluoroisopropanol group is highly transparent at 157 nm. This is illustrated by the absorbance data in Fig. 4 showing that incorporation of the fluoroalcohol side chain actually improves the transparency of the already highly transparent TFE/norbornene copolymer.

The TFE/NB-F-OH dipolymer is soluble in aqueous base but its rate of dissolution is too slow for a practical photoresist so some *t*-BuAc is used as the solubility switch. However, the required level of acrylate is significantly reduced by the presence of the fluoroalcohol groups so that the terpolymers have absorbance values in the range of 1.0–2.5, depending on acrylate content.

These TFE/NB-F-OH/*t*-BuAc terpolymers have glass transition temperatures in the range of 140–150 °C and weight average molecular weight of around 10,000, a suitable value for balancing mechanical properties and dissolution rate. The polymers are readily soluble in moderately polar organic solvents such as acetone, 2-heptanone, THF and chloroform and insoluble in hexane and methanol.

### 2.2. Polymerization technology

There are several key issues with respect to the polymerization technology for preparation of the fluorinated photoresist polymers including reactivity ratios of the monomers and reactivity of the substituted norbornene monomer. The terpolymers are prepared by free-radical solution polymerizations using a peroxydicarbonate initiator and a hydrofluorocarbon solvent. TFE and norbornene have a strong tendency to alternate as expected for the combination of a highly fluorinated olefin with a hydrocarbon olefin [14] so control of polymer composition is simple with the dipolymer. The situation is made more complex by the acrylate termonomer as illustrated by a series of batch polymerizations of TFE, norbornene and *t*-BuAc (Table 2). *t*-BuAc feed concentrations of 0, 2, 5, 10 and 20 mol% were used and each recipe was run to low and high conversion by varying the amount of initiator. The results at low conversion show that the acrylate content in the polymer is 2.8–4.0 times greater than that present in the monomer mixture. At high conversion, the overall polymer composition is nominally closer to the monomer feed composition but this suggests the formation of a very inhomogeneous blend during the

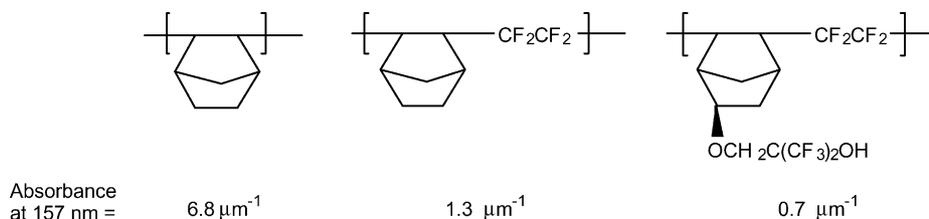


Fig. 4.

Table 2  
Batch polymerizations of TFE, norbornene and *tert*-butyl acrylate (*t*-BuAc)<sup>a</sup>

| Entry | Monomer feed (mol%) |     |                | Initiator <sup>b</sup><br>(mol%) | Yield (%) | Polymer composition (mol%) <sup>c</sup> |     |                | Mn   |
|-------|---------------------|-----|----------------|----------------------------------|-----------|---|-----|----------------|------|
|       | TFE                 | NBE | <i>t</i> -BuAc |                                  |           | TFE                                     | NBE | <i>t</i> -BuAc |      |
| 1     | 70                  | 30  |                | 0.25                             | 31        | 52                                      | 48  |                | 6700 |
| 2     | 70                  | 30  |                | 1.0                              | 89        | 70                                      | 30  |                | 4900 |
| 3     | 68                  | 30  | 2              | 0.25                             | 26        | 49                                      | 43  | 8              | 5200 |
| 4     | 68                  | 30  | 2              | 1.0                              | 91        | 64                                      | 32  | 4              | 4500 |
| 5     | 65                  | 30  | 5              | 0.25                             | 24        | 37                                      | 44  | 19             | 6200 |
| 6     | 65                  | 30  | 5              | 1.0                              | 89        | 56                                      | 40  | 5              | 4300 |
| 7     | 60                  | 30  | 10             | 0.25                             | 27        | 28                                      | 35  | 37             | 6300 |
| 8     | 60                  | 30  | 10             | 1.0                              | 87        | 53                                      | 35  | 13             | 5600 |
| 9     | 50                  | 30  | 20             | 0.25                             | 29        | 16                                      | 28  | 56             | 9900 |
| 10    | 50                  | 30  | 20             | 1.0                              | 63        | 42                                      | 40  | 18             | 6400 |

<sup>a</sup> Polymerizations in a 200 ml shaker tube at 50 °C using 1,1,2-trichlorotrifluoroethane solvent.

<sup>b</sup> Initiator is di-(*tert*-butylcyclohexyl)peroxydicarbonate (Perkadox<sup>®</sup> 16N).

<sup>c</sup> Compositions determined by <sup>13</sup>C NMR.

course of the polymerization as the more reactive acrylate is depleted. Thus, a semibatch process, described in Section 4, was developed. Polymerization is initiated with an acrylate-lean mixture in the reactor. Initiator and a more acrylate-rich monomer mixture are fed continuously to the reactor during the course of the polymerization. This process has now been scaled-up to the production of multikilogram batches.

A second issue regarding polymerization is the reactivity of substituted norbornenes. Although norbornene itself polymerizes with TFE as described [15], we found that the available 5-substituted norbornenes were substantially less reactive, a situation previously observed with other norbornene copolymerizations [16]. We then discovered that the polymerizability of 5-substituted norbornenes depends critically on the stereochemistry of the substituent at the 5-position. This is illustrated by the data in Table 3, which

compares copolymerization of TFE with a commercially available mixture of endo- and exo-norborn-2-en-5-yl acetates and a copolymerization with the pure exo-acetate [17]. The latter is seen to give substantially improved conversion and higher molecular weights.

The available substituted norbornenes are generally a mixture of endo- and exo-isomers, mostly endo, due to their syntheses by cycloadditions to cyclopentadiene. The need for a norbornene with a purely exo-fluoroalcohol substituent at the 5-position led to the synthesis shown in Fig. 5. The known [17] exo-acetate, prepared by addition of acetic acid to norbornadiene, is hydrolyzed to the exo-alcohol. The sodium salt of the alcohol then reacts readily with hexafluoroisobutylene oxide [18] to afford NB-F-OH which polymerizes with TFE to give polymer with acceptable conversion and molecular weight.

### 2.3. Tricyclononene copolymers

Could incorporation of additional fluorine result in further enhancement in polymer properties? As noted above, the cyclic hydrocarbon monomer and TFE have a strong tendency to alternate so simply increasing the TFE content in the backbone would be difficult. In addition, runs of TFE are likely to decrease transparency [19] and, if too long, could

Table 3  
Copolymerization of TFE and norborn-2-en-5-yl acetates<sup>a</sup>

| Norbornenyl acetate | Conversion (%) | Mn/Mw     |
|---------------------|----------------|-----------|
| Endo/exo mixture    | 8              | 2100/3100 |
| Exo                 | 33             | 3200/8300 |

<sup>a</sup> Batch copolymerization of a 60/40 mixture of TFE/norbornenyl acetate in Solkane<sup>®</sup> 365 solvent using 0.8 mol% Perkadox<sup>®</sup> 16N initiator.

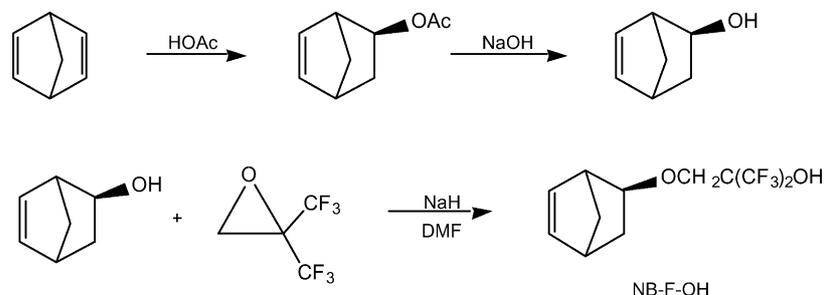


Fig. 5.

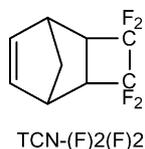


Fig. 6.

lead to undesirable crystallinity in the polymer. Adding fluorine to the norbornene ring in proximity to the double bond would have a negative effect on polymerizability.

One approach which we have begun to explore is through the development of a family of fluorinated tricyclononene monomers. The prototypical example is the monomer TCN-(F)2(F)2 (Fig. 6) readily prepared by cycloaddition of TFE and norbornadiene.

In practice, we found that this tricyclononene monomer copolymerizes as well or better with TFE than does norbornene, presumably due to the fact that the four membered ring is on the exo face of the bicycloheptene system and the fluorines are sufficiently remote from the double bond. Interestingly, the TFE/TCN-(F)2(F)2 copolymer has a significantly higher glass transition temperature (228 °C versus 150 °C) and is more transparent ( $A = 0.69 \mu\text{m}^{-1}$  versus  $1.3 \mu\text{m}^{-1}$ ) than the TFE/norbornene copolymer. We are currently exploring copolymers of TFE with various functionalized TCN monomers for this application.

### 3. Conclusions

Fluoropolymers already have significant applications in the semiconductor industry, mainly as materials for handling the corrosive and/or high purity chemicals used for making computer chips. These applications are mainly passive and protective and derive from the exceptional chemical and thermal stability of highly fluorinated polymers. It now seems likely that fluoropolymers will play a more active role in chip manufacture by functioning as the photoresist binder polymer in the actual process of chip manufacture. The new role for fluorine chemistry comes from the impact of fluorination on the absorption spectrum of materials at very short wavelengths and from the effect of fluorination of acidity of a key functional group. However, careful selection of comonomers and polymerization conditions are required for achieving the desired balance of polymer properties.

### 4. Experimental details

GPC data were obtained on Waters Alliance 2690 instrument with a model 410 refractometer using THF as the mobile phase at  $1 \text{ ml min}^{-1}$ . Column set consisted of three PL gel  $5 \mu\text{m}$  particle size columns, two mixed C and one 500A. Column temperature was 35 °C and data is reported versus polystyrene standards. DSC and TGA data were obtained on TA Instruments model 2920 MDSC and model

2950 TGA, respectively.  $T_g$  is reported as the midpoint in the deflection on a second heat from  $-25$  to  $275$  °C at a heating rate of  $20$  °C  $\text{min}^{-1}$ . Thermal decomposition temperature is reported as the temperature of 10 wt.% loss under nitrogen by TGA with a heating rate of  $20$  °C  $\text{min}^{-1}$ .

Exo-5-norbornen-2-ol was prepared by addition of acetic acid to norbornadiene, followed by alkaline hydrolysis. TCN-(F)2(F)2 was prepared by reaction of tetrafluoroethylene (1 equivalent) and norbornadiene (1.25 equivalents) at 180 °C for 8 h in a sealed metal pressure vessel. The product was purified by spinning band distillation to provide pure cuts of the tricyclic comonomer, having a boiling point of 67 °C at 45 mm.

#### 4.1. Synthesis of NB-F-OH

A dry round bottom flask equipped with mechanical stirrer, addition funnel and nitrogen inlet was swept with nitrogen and charged with 19.7 g (0.78 mol) of 95% sodium hydride and 500 ml of anhydrous DMF. The stirred mixture was cooled to 5 °C and 80.1 g (0.728 mol) of exo-5-norbornen-2-ol was added dropwise so that the temperature remained below 15 °C. The resulting mixture was stirred for 1/2 h. Hexafluoroisobutylene oxide [20] (131 g, 0.728 mol) was added dropwise at room temperature. The resulting mixture was stirred overnight at room temperature. Methanol (40 ml) was added and most of the DMF was removed on a rotary evaporator under reduced pressure. The residue was treated with 200 ml water and glacial acetic acid was added until the pH was about 8.0. The aqueous mixture was extracted with  $3 \times 150$  ml ether. The combined ether extracts were washed with  $3 \times 150$  ml water and 150 ml brine, dried over anhydrous sodium sulfate and concentrated on a rotary evaporator to an oil. Kugelrohr distillation at  $0.20$ – $0.27 \times 10^{-4}$  MPa and a pot temperature of 30–60 °C gave 190.1 g (90%) of product.  $^1\text{H NMR}$  ( $\delta$ ,  $\text{CD}_2\text{Cl}_2$ ) 1.10–1.30 (m, 1H), 1.50 (d, 1H), 1.55–1.65 (m, 1H), 1.70 (s, 1H), 1.75 (d, 1H), 2.70 (s, 1H), 2.85 (s, 1H), 3.90 (d, 1H), 5.95 (s, 1H), 6.25 (s, 1H). Anal. Calcd. for  $\text{C}_{11}\text{H}_{12}\text{F}_6\text{O}_2$ : C, 45.53; H, 4.17; F, 39.28. Found: C, 44.98; H, 4.22; F, 38.25.

#### 4.2. Synthesis of a TFE/NB-F-OH/*t*-BuAc terpolymer

A metal pressure vessel of approximately 1 l capacity was charged with 206.6 g NB-F-OH, 4.80 g *t*-BuAc and 75 ml of Solkane 365 mfc (1,1,1,3,3-pentafluorobutane). The vessel was closed, cooled to  $-15$  °C and pressured to 400 psi with nitrogen and vented several times. The reactor contents were heated to 50 °C and TFE was added from a reservoir to a pressure of 2.21 MPa. A regulator was set to maintain pressure at 2.21 MPa throughout the polymerization. A solution of 18.45 g Perkadox<sup>®</sup> 16N which had been dissolved in 100 ml of methyl acetate and then diluted to 200 ml with Solkane 365 mfc was added at  $6.0 \text{ ml min}^{-1}$  for 6 min and then at  $0.24 \text{ ml min}^{-1}$  for 8 h. Simultaneously, with the start of initiator feed, a solution of 203.9 g NB-F-OH and

30.0 g *t*-BuAc diluted to 250 ml with Solkane 365 mfc was added at 0.28 ml min<sup>-1</sup> for 12 h. After 16 h reaction time, the vessel was cooled to room temperature and vented to 1 atm. The reaction solution was recovered using additional Solkane 365 mfc to rinse. The combined reactor solution and rinse was concentrated on a rotary evaporator to a thick oil. The oil was added in a thin stream to 5.1 l of vigorously stirred heptane. The resulting slurry was stirred for 30 min, then filtered. The solid was washed with heptane and allowed to dry on the filter for 90 min (unreacted NB-F-OH is readily recovered by evaporation of the filtrate and distillation). The solid polymer (219.6 g) was dissolved a mixture of 94 ml THF and 350 ml Solkane 365 mfc and this solution was added slowly to 11 l heptane. The resulting slurry was stirred for 35 min, then filtered. The solid was washed with 300 ml heptane and dried in a vacuum oven at 80 °C to give 162.4 g of polymer. The polymer composition was determined to be 32% TFE, 46% NB-F-OH and 22% *t*-BuAc by <sup>13</sup>C NMR (in CD<sub>2</sub>Cl<sub>2</sub>) using integrals of peaks at 64.6 (OCH<sub>2</sub> of NB-F-OH), 105.9–132.7 (CF<sub>2</sub> of TFE and CF<sub>3</sub> of NB-F-OH), and 73.6–87.8 (one quaternary carbon of *t*-BuAc and two carbons from NB-F-OH). <sup>19</sup>F NMR (δ, CDCl<sub>3</sub>) -76 to -77 (CF<sub>3</sub>), -95 to -125 (CF<sub>2</sub>). GPC: Mn = 6900, Mw = 9900, Mw/Mn = 1.44. DSC: T<sub>g</sub> = 150 °C, TGA: T<sub>d</sub> = 292 °C. Anal. Found: C, 46.51; H, 4.51; F, 34.35.

#### 4.3. Synthesis of TFE/TCN-(F)2(F)2 copolymer

A 200 ml stainless steel pressure vessel was charged with 46.1 g of TCN-(F)2(F)2, 75 ml of 1,1,2-trichlorotrifluoroethane and 1.0 g of Perkadox<sup>®</sup> 16N. The vessel was closed, cooled in dry ice, evacuated and charged with 36 g of tetrafluoroethylene. The vessel was then agitated with its contents at 50 °C for 18 h. The vessel was cooled to room temperature and vented to one atmosphere. The translucent, gel-like solution was removed from the vessel using additional 1,1,2-trichlorotrifluoroethane to rinse. The mass was allowed to air-dry. The polymer was dissolved in tetrahydrofuran and precipitated into excess methanol. The solid was dried in a vacuum oven at 85 °C giving 18.0 g of white polymer; GPC: Mn = 9400, Mw = 13,100; Mw/Mn = 1.40. DSC: T<sub>g</sub> = 228 °C. <sup>19</sup>F NMR -95 to -122 (multiplets, 4F from TFE and 2F from NB-TFE), -124.4 (dd, 2F from NB-F-OH). From integration of the spectrum, the polymer composition

was calculated to be 53% TFE and 47% NB-TFE. The absorbance at 157 nm was found to be 0.69 μm<sup>-1</sup> from measurements on spin cast films of 88.4 and 102.9 nm thickness. Anal. Found: C, 46.26; H, 2.90; F, 49.80.

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